AVA300 nmr (1D)user guide

20130305 L-2

Note : The magnetic object do not be put over the yellow line of magnet ； Do not push magnet.

(A) Login name:_____

(1) log on, type user : nmr1

(2) click TOPSPIN 2.1

(3) click （open the BSMS panel）

(B) Put the sample

(4) clean the tube and spinner (inside and outside)

(5) put tube in spinner (the tube height don't over 1.8 cm)

(6) click BSMS panel Main / SAMPLE / LIFT wait to change to green and hear the air voice

(7) put the tube in magnet, click Main / SAMPLE / LIFT to close the air

(C) nmr

1. Create a new file :

(8) type **edc** Enter

NAME	****** (don't use special character ,such as： * ? > < ; & ! [ ] | \ ' " ` ( ) { }. Don't use - or + at first word)
EXPNO	1 ~999□ （experiment number）
PROCNO	1
DIR	/x
USER	**cclin, kllu (small letter)**
Solvent	Select the solvent
Experiment	Select the standard file 1D_1H_qnp 1D_13C_dh_qnp 1D_19F_dh_qnp 1D_31P_dh_qnp 1D_DEPT45_qnp 1D_DEPT90_qnp 1D_DEPT135_qnp [for av3-400 nmr : st_1D_1H_bbfo, st_1D_13C_dh_bbfo, st_1D_31P_dh_ bbfo , st_1D_19F_dh_ bbfo st_2D_COSY_ bbfo , st_2D_HMBC_ bbfo st_2D_NOESY_ bbfo , st_2D_TOCSY_ bbfo]
TITLE	Set title
Note or title	

（file path: D:\DATA\USER\NMR******\EXPNO）

(9) type **i i** Enter

(10) type **rsh shim.1** **(**or **rshs)** Enter (qnp probe)

[for av3-400 nmr : (10) type **rshs**]

(11) click (or type **lockdisp** Enter) display lock panel

(12) type **lock** Enter, choice CDCL3, DMSO, ...OK

p.s. If you can't lock sample, click Main / LOCK / On/Off

(13) click BSMS panel the Main / SAMPLE / SPIN

（check rate: click Sample & Level / SAMPLE SPIN / rate measure）

[for av3-400 nmr : (13) you don't need to click SPIN]

2. Shimming

(14) click Main / SHIM / Z

click stepsize to change the level for every step（as the crude or fine tune）

click Main / VALUE / step+ 、 step- till lock level line is uppermost。

Repeat to click Z^2, Z （if lock level line over the range of window ,use BSMS panel

change Main / LOCK / Gain or Power make lock level line is at upper 4 line）

[for av3-400 nmr : (14) type **topshim**]

3. Acquisition :

(15) type **getprosol** Enter

(16) type **rga** Enter (type **rg** Enter)

(17) type **ns** Enter set the multiple of 4 Enter

(18) type **zg** Enter (start run the experiment)

tr Enter (transfer part of dara)

for ^{1}H : type **ft** Enter

for 13C or 31P: type **lb** Enter **3** Enter ; type **ef** Enter

(19) wait for zg finished (or type **halt** to stop experiment) 。

Run ^{13}C or ^{31}P (the same sample)

Repeat (8)(9)(15) ~ (19)

[for av3-400 nmr : Repeat (8)(9) type **atma** (15) ~ (19)]

(D) logout

(20) type **halt** Enter (make sure the nmr experiment is already stop)

(21) click BSMS panel the upper Main / LOCK / on/off ; Main / SAMPLE / SPIN

☆(a) check ：Sample & Level / SAMPLE SPIN / rate measure =0

☆(b) check ：lock level line display as sine wave

[for av3-400 nmr : (21) click BSMS panel the upper Main / LOCK / on/off]

(22) click Main / SAMPLE / LIFT ; take nmr tube out ; click Main / SAMPLE / LIFT

(23) click your data file top right corner x turn off your data file

(24) click top right corner x turn off Topspin interface , you don't need to log off

PC:

(E) Processing

(25) click View /Browser Pnael on/off to find your own data

(26) for 1H : type **ft** Enter

for 13C or 31P : type **lb** Enter **3** Enter type **ef** Enter

Phase:

auto phase correction (27) ; **manual phase correction** (28)

(27) type **apk** Enter to auto phase correction.

(28) manual phase correction, move mouse to click , into phase panel (the window upper-leftside has yellow color), move mouse to 0 hold mouse to move up side down, move mouse to 1 hold mouse to move up side down ,till the phase is correct. Then click up-right to save.

Baseline Correction :

(29) type **abs n**

Set Standard Peak :

(30) click , into standard peak panel (the window upper-left corner has yellow color), move the mouse to solvent peak click left button key in the chemical shift OK 。

Set Minimum Intensity :

(31) click , into intensity panel(the window upper-left corner has yellow color), when is green, move mouse to spectrum hold left button to drag, make the square cover the peak then will show the peak picking or click then move the mouse to square edge hold left button to drag, it will change the square area, (you also can make the many small square to cover the peak) Then click to save 。

Integration :

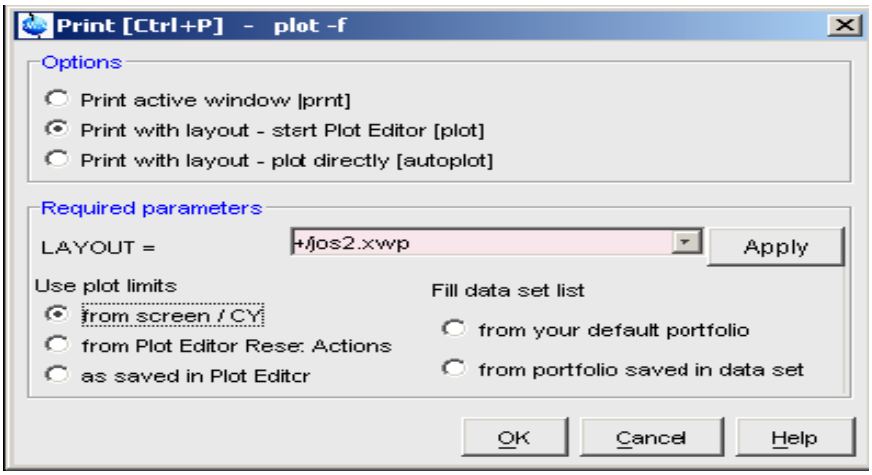
(32) click , into integrated panel(the window upper-left corner has yellow color), move

mouse to spectrum hold left button to drag from peak left side to right side to expand spectrum.

Then click □ to let □ change to green color , when you move mouse to spectrum then hold left button to drag from peak left side to right side, peak will be integrated. Then click up-right □ to save。

Plot :

(33) click □ will display



(34) LAYOUT

for 1H : choice +/1D_H.XWP or

for 13C or 31P : choice +/1D_X.XWP click OK into the xwinplot panel

(35) change the spectrum range integration parameter etc make mouse arrow to the spectrum or parameter click left button then the spectrum parameter corner had 8 green spot

at this moment , click right button will show

Edit : set the plot spectrum range , integrate, preak level…

1D/2D Edit : set spectrum and integrate level

(36) click □ to print out your spectrum

p.s.

1. change sweep range : before zg ; type sw Enter to change the value.

3.When you copy file to process by Topspin, you need to create some sub folder

<dir>\data\<user>\ nmr \ <your dataset name> , then put the file to <your dataset name>

spooler：

type **qu** (display queue time) **edqu** (edit queue time)

	create file		Move to end of top
	Open file		Hold left-click withdraw then move up and down to change peak height
	Save file		Move to end of down
	Print		switch Hz / ppm unit
	copy		Peak height *2
	paste		Peak height /2
	Phase mode		Peak height *8
	Set reference mode		Peak height /8
	baseline correction mode		Hold left-click withdraw to move up and down change peak height
	peak picking mode		Set peak height
	Integration mode		Set range of spectrum
	Overlape spectrum		Full spectrum
	Spectrum move right		Increase range of spectrum
	Hold left-click withdraw to move right and left change range of spectrum		Reduce range of spectrum
	Spectrum move left		Hold left-click withdraw then move up and down to change range of spectrum
	Spectrum move to end-of- left		Set edge of spectrum
	Spectrum move to end-of- right		Go back last range of spectrum